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Dynamic optimization and robust control of batch crystallization

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Abstract

Crystallization is used to produce vast quantities of materials. Batch crystallization is widely practiced for high-value products. Batch crystallizers are desired to operate at an optimal trajectory to produce a desired product quality and crystal size distribution (CSD). In this work the dynamic optimization via nonlinear programming techniques and the robust control of a non-isothermal batch crystallizer with two practical robust control approaches is addressed: (i) modeling error compensation, and (ii) integral high order sliding mode control. The controller designs are based on the reduced-order model representation of the population balance equation resulting after the application of the method of moments. Numerical simulations show good closed-loop performance and robustness properties.

Keywords: Batch processes; crystallization; robust control; sliding mode control; modeling error compensation

1. Introduction

Crystallization, one of the oldest of unit operations, is used to produce vast quantities of materials, including sodium chloride, sodium and aluminum sulphates and sucrose. Crystallization is also a key operation in the freeze-concentration of fruit juices, the desalination of sea water, the recovery of valuable

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materials such as metal salts from electroplating processes, the production of materials for the electronic industries and in biotechnological operations such as the processing of proteins. [1, 2].

Crystallization can be carried out in batch, fed-batch, and continuous crystallizers. Continuous, steady-state operation is not always the ideal mode for the operation of crystallization processes, and batch operation often offers considerable advantages such as simplicity of equipment and reduced encrustation on heat-exchanger surfaces. Whilst only a batch crystallizer can, in certain cases, produce the required crystal form, size distribution, or purity, the operating costs can be significantly higher than those of a comparable continuous unit, and problems of product variation from batch to batch may be encountered. Semi-continuous crystallization processes which often combine the best features of both batch and continuous operation [1,2].

Batch crystallizers are desired to operate at an optimal trajectory to produce a desired product quality and crystal size distribution (CSD) [2-6]. The optimization of batch processes has attracted attention in recent years because, in the face of growing competition, it is a natural choice for reducing production costs, improving product quality, meeting safety requirements and environmental regulations [5-9].

Because open-loop implementations of optimal profiles are very sensitive to modeling errors and disturbances, control policies are required to maintain the specific product quality and to suppress the influence of process disturbances. Within the control theory framework, the problem can be formulated as a tracking problem where the computed optimal profile becomes the reference trajectory for a feedback controller. Challenges in controlling crystallization includes significant uncertainties associated with their kinetics, phenomenological effects are difficult to characterize, and the fact that crystallization processes are highly nonlinear, and are modeled by coupled nonlinear algebraic integro-differential partial equations [3,4, 9-11]. Robust feedback control seems to be essential for crystallization processes as it permits to deal with model uncertainties related to model parameters and model reduction for control design purposes. From the point of view of controlling a crystallizer the main quality criteria are the properties of the produced crystals, first of all the size-distribution and the mean size [3,4].

In this work, the dynamic optimization via a nonlinear programming technique, and the robust control of batch crystallizers, with two practical robust control approaches is addressed: (i) modeling error compensation, and (ii) integral high order sliding mode control. Although the crystallization control literature is vast, to the authors' knowledge there is no general framework for the synthesis of practically implementable robust feedback controllers for batch crystallization processes. The controller designs are based on the reduced-order model representation of the population balance equations resulting after the application of the method of moments. Numerical simulations show good closed-loop performance and robustness properties.

This work is organized as follows: In Section 2 the class of batch crystallizers are presented, including the case study. In Section 3 the dynamic optimization formulation is presented. In Section 4, robust control designs are presented. Numerical results are presented and discussed in Section 5. Finally in Section 6 some concluding remarks are presented.

2. The class of batch crystallizers

In this section we describe the case study of a non-isothermal batch crystallizer and a general class of crystallization batch models, based on reduced models via the moment approach. For the sake of completeness we also provide some generalities of crystallization processes.

2.1. Crystallization brief overview.

Crystallization is a separation process that brings about the formation of solid crystals from a fluid phase, namely vapor, solution or melt, wherein the solubility characteristics of certain materials are exploited to produce particles of a very high purity. Main elements for crystallization are a driving force, the operation between metastable and labile zones, and nucleation and growth stages [1,2].

Driving forces can be generated through a variety of means, including cooling or heating to reduce or increase the system temperature, evaporating solvent, evaporative (flash) cooling, inducing a chemical reaction, adjusting pH, salting out through the addition of a nonsolvent, direct-contact cooling with a refrigerant, or some other means. Metastable and labile zones refers to supersaturated solutions in which the spontaneous deposition of the solid phase, in the absence of crystallizing solid material, will or will not occur, respectively. A supersaturated solution is not in equilibrium. In order to relieve the supersaturation and move towards equilibrium, the solution crystallizes. Once crystallization starts, the supersaturation can be relieved by a combination of nucleation and crystal growth. Nuclei are necessary for the deposition of solute material on the crystal lattice surface (growth). The process of nucleation involves the formation of new crystals in a crystallizing environment. Nuclei growth by the addition of solute molecules from the supersaturated solution [1,2]. All of these modes of operation can be implemented in either a batch or a continuous process. In addition, two or more of the modes may be combined to enhance the product yield.

A typical mathematical model of a crystallizer consists of the population balance equation for crystals and the balance equations for solvent and crystallizing substance, the enthalpy balance, and the equations describing the variation of the equilibrium saturation concentration [1-4]. The population balance is in fact a distributed mass balance for a solid or dispersed phase, and is linked to the liquid or continuous phase component mass balances via the crystallization kinetics [1-4. 10].

2.2. Case study: Seeded batch non-isothermal crystallizer.

Consider the seeded batch crystallizer of potassium sulfate studied by Shi et al. [9]. In the seeded crystallizers, the relationship between the mass fraction of the seeds and that of the newly generated nuclei has a significant effect on the product quality. The process model of a seeded batch crystallizer has the following form [5,9],

$$\begin{aligned}
&\frac{\partial f(L,t)}{\partial t} + G(t)\frac{\partial f(L,t)}{\partial L} = 0 \qquad\qquad f(L,0) = \frac{B(t)}{G(0,t)} \\
&\frac{dC}{dt} = -3\rho_c k_v G(t)\int_0^\infty f(L,t)L^2 dL \\
&\frac{dT}{dt} = -3\frac{\Delta H}{C_p}\rho_c k_v G(t)\int_0^\infty f(L,t)L^2 dL - \frac{UA}{MC_p}(T(t) - T_j(t))
\end{aligned} \tag{1}$$

where ρ is the density of crystals, k_v is the volumetric shape factor, U is the overall heat-transfer coefficient, A is the total heat-transfer surface area, M is the mass of solvent in the crystallizer, c_p is the heat capacity of the solution, T_j is the jacket temperature, and H is the heat of reaction. The nucleation rate, $B(t)$, and the growth rate, $G(L, t)$, are given by [5,9],

$$B(t) = k_v \exp(-E_b / RT)\left(\frac{C - C_s(t)}{C_s(t)}\right)^b \int_0^\infty f(L,t)L^3 dL$$
$$G(t) = k_g \exp(-E_g / RT)\left(\frac{C - C_s(t)}{C_s(t)}\right)^g \quad (2)$$

where E_b is the nucleation activation energy, E_g is the growth activation energy, b and g are exponents relating nucleation rate and growth rate to supersaturation [5,9]. The saturation concentration of the solution C_s, and the zero-size boundary condition for model (1) are [5,9],

$$C_s(T) = 6.29\times10^{-2} + 2.46\times10^{-3}T - 7.14\times10^{-6}T^2$$

The population balance model is not appropriate for synthesizing simple model-based, low-order feedback control laws due to its distributed parameter nature. Following the moments reduction technique [1,2,5], the following reduced-order moments model is obtained as follows,

$$\mu_i = \int_0^\infty f(L,t)L^i(t)dL \qquad \mu_i = \mu_i^n + \mu_i^s$$
$$\frac{d\mu_0^n}{dt} = B(t) \qquad \frac{d\mu_0^s}{dt} = cte$$
$$\frac{d\mu_i^n}{dt} = iG(t)\mu_{i-1}^n(t) \qquad \frac{d\mu_i^s}{dt} = iG(t)\mu_{i-1}^s(t)$$
$$\frac{dC}{dt} = -3\rho_c k_v G(t)\mu_2 \quad (3)$$
$$\frac{dT}{dt} = -3\frac{\Delta H}{C_p}\rho_c k_v G(t)\mu_2 - \frac{UA}{MC_p}(T(t) - T_j(t))$$

where μ_i (i = 0, 1, 2, 3) are dimensionless moments of the crystal size distribution. It can be demonstrated that the total number of crystals, the total length, the total area, and the total volume of crystals, all in a unit of sample volume, can be evaluated from the zeroth, first, second, and third moments of the population density function. Simulation of dynamic behaviour of the crystallizer was performed with process parameters given in [5,9].

2.3. The class of crystallizer models.

For the dynamic optimization and control design purposes, we consider the following class of mathematical model for continuous crystallizers,

$$\frac{dy}{dt} = f_1(y,z) + g(y,z)u + \psi(y,z) + \phi(t)$$
$$\frac{dz}{dt} = f_2(y,z) \quad (4)$$

where $\psi(y, z)$ unmodeled dynamics, and $\phi(t)$ external perturbations, $f_1(y,z) \in R$, $f_2(y,z) \in R^{n-1}$, and $g(y,z) \in R$, are smooth functions of their arguments, $y \in R$, is the measured output of the system, $z \in R^{n-1}$, is the internal state, and u is the control variable or control input. It is not hard to see that several published

models of batch crystallizers can be described by model (4) using the moments reduction technique for the PSD [3-6, 9-11]. It should be noted that representation (4) will be used as it stands for control design purposes. For dynamic optimization purposes, model (4) without model uncertainties and external perturbations will be considered.

3. Dynamic optimization for batch crystallization

In this section the generic problem formulation for the dynamic optimization of batch crystallization processes is described. The dynamic optimization model for the case study is also presented.

3.1. Dynamic optimization model formulation.

A typical dynamic optimization setup requires a dynamic process model, definition of existing operating procedures, an objective function, operating constraints, and a set of optimization variables [7,12]. For the batch crystallization problem, the dynamic optimization model formulation is then given as follows [5,9],

$$\begin{aligned} &\min_{t_f,u(t)} J = \phi(x(t_f)), \\ &s.t. \qquad \frac{dx}{dt} = \Phi(x,u) \qquad\qquad x(0) = x_0 \\ &S(x,u) \le 0, \\ &T(x(t_f)) \le 0 \end{aligned} \tag{5}$$

where J is the scalar performance index to be minimized, x, the n-dimensional vector of states with known initial conditions x_0, u, the control variable, S stands for state constraints and input constraints, and T stands for terminal constraints. Φ is a smooth vector function, ϕ, a smooth scalar function representing the terminal cost, and t_f the final time that is finite but can be either fixed or free. This problem formulation is quite general.

From the point of view of the optimization in crystallization, the main quality criteria are the properties of the produced crystals, first of all the PSD and the mean size [1-4]. However, due the complexity of the measurement, mathematical treatability and control of the PSD, most authors have been addressed both the dynamic optimization and the control of crystallization processes via objective functions related to the PSD such as the second and third moment, solute concentration and crystallizer temperature. Common objectives for the CSD at the end of the batch are [1-4]: (i) minimize the amount of nucleus-grown crystals, (ii) maximize the average size of the total crystals, (iii) minimize the variation in size of the total crystals, (iv) grow the seed crystals as large as possible. These objectives can be formulated as a function of both low and high order moments of the CSD.

The control variable for the dynamic optimization, which be the control input for control design purposes can be selected from a number of options. For instance, carefully selected seed crystals are sometimes added to a crystallizer to control the final product crystal size. The product crystal size from a batch crystallizer can also be controlled by adjusting the rates of cooling or evaporation. Indeed, temperature profile affects supersaturation profile which has strong influence on the CSD. Thus, optimization of temperature profiles is of great importance. Semi-batch crystallization, on the other hand, can be controlled by suitable variations of feed conditions [3-7].

3.2. Case study: dynamic optimization model formulation.

The dynamic optimization model for case study is given as follows [5,9],

$$\begin{aligned} &\min_{tf,u(t)} J = \phi(x(t_f)) = \mu_3^n(t_f) - \mu_3^s(t_f), \\ &s.t. \qquad Eq.(3) \qquad\qquad x(0) = x_0 \\ &C_s \le C \le C_m, \\ &T_{\min} = 303K \le T \le T_{\max} = 323K, \\ &\left|\frac{dT}{dt}\right| \le K \end{aligned} \tag{6}$$

In batch crystallization processes, a large volume of seeded crystals favours product quality. On the other hand, fine crystals obtained from nucleation should be kept in possible lower limit as they may cause difficulties in downstream operations e.g. filtration and drying. Thus, the aim of a dynamic optimization is to maximize the total volume of seeded crystals (μ_3^s) whereas keeping the total volume of fine crystals (μ_3^n) small [5,9]. The constant, k, is the maximum gradient of the crystallizer temperature chosen to be zero to avoid the increase of the temperature, and the final batch time, t_f, is 30 min. μ_3^s. Control variable is the jacket crystallizer temperature. The dynamic optimization problems leads to a optimal crystallizer temperature profile.

3.3. Solution of the dynamic optimization model.

The optimization problem is solved in *Matlab* environment using *dynopt* package, which is well suited for the solution of constrained optimization problems by means of non-linear programming (NLP) algorithms [13,14]. *Dynopt* first uses the method of orthogonal collocations on finite elements to transform ordinary differential equations to algebraic equations by parameterization of the state and input trajectories. The optimal control problem is then converted to an NLP problem via simultaneous optimization approach and solved using a sequential quadratic programming algorithm, which is suitable to solve medium-scale optimization problems subject to inequality constraints [13,14].

4. Robust control of batch crystallizers

In this section the control problem of batch crystallization processes is introduced. Robust control approaches are also described.

4.1. The control problem.

Once formulated and solved the dynamic optimization in batch crystallization problems, is obtained an optimal operation policy in terms of crystallizer control variable for maximizing a given objective function. The resulting optimal policy is implemented as set-point, denoted as y_{ref}, for closed-loop control studies. Following this ideas, in this work the control problem consists of the regulation or tracking, of the optimal policy y_{ref}, obtained from a dynamic optimization problem, via manipulation of the corresponding control variable u.

The control problem description is completed by the following assumptions:

- A1: The measurement of the variable to be controlled, denoted as y, is available for control design purposes.

- A2: Nonlinear functions *f(y,z)* and *g(y,z)* for the class of crystallizer models given (8) are uncertain, and can be available rough estimates of these terms.
- A3: The class of crystallizer models given (8) is affected by unmodeled dynamics $\psi(y,z)$ and external perturbations $\varphi(t)$.

4.2. Modeling error compensation approach.

The underlying idea behind MEC control designs is to lump the input-output uncertainties into a term, which is estimated using a high-gain observer and compensated via a suitable inverse feedback function with a desired closed-loop behaviour. MEC approach was introduced by Sun et al. [15] for linear systems and further extended by Alvarez-Ramirez [8, 16] for a class of non-linear linearizable systems with exponential and finite-time convergence, respectively.

Consider the class of continuous crystallizers described in Section 2, under assumptions A1 and A2,

$$\begin{aligned} \frac{dy}{dt} &= \eta(t) + \bar{g}_1 u(t) \\ \eta(t) &= f_1(y,z) + [g_1(y,z) - \bar{g}_1(y,z)]u + \psi(y,z) + \varphi(t) \end{aligned} \tag{7}$$

where η is the modeling error function, and the term $g_1(y,z)$ in (7) is a rough estimate of its real value. A reduced order observer is introduced to estimate the modeling error function in (7), as follows [16],

$$\begin{aligned} \frac{dw}{dt} &= -\bar{g}_1 u - \bar{\eta} \\ \bar{\eta} &= \tau_e^{-1}(w + y) \end{aligned} \tag{8}$$

such that,

$$\frac{dy}{dt} = \bar{\eta}(t) + \bar{g}_1 u(t) \tag{9}$$

an inverse-dynamics with finite-time convergence is introduced as follows [8],

$$u = -\bar{g}_1^{-1}(\bar{\eta}(t) + \tau_c^{-1}|e|^{-1/n}) \tag{10}$$

where $e = y - y_{ref}$, is the regulation or tracking error, τ_e, τ_c and n are estimator and control design parameters, which can be selected as follows $t_f > \tau_c >> \tau_e$ and n an odd number. The closed-loop performance is given as [8],

$$\frac{de}{dt} = -\tau_c^{-1}\|e\|^{-1/n} \tag{11}$$

In particular, for n=3, the solution of (11) is,

$$e = sign(e_0)[|y_0| - \tau_c^{-1} t/3]^3 \tag{12}$$

such that a finite-time convergence with $t = 3\tau_c^{-1}|y_0|^{1/3}$ is guaranteed [8].

4.3. High-order sliding mode control.

Sliding mode control techniques have long been recognized as a powerful robust control method [17-19]. The design of a SMC involves designing of a sliding surface that represents the desired stable dynamics and a control law that makes the designed sliding surface attractive. The conventional SMC design approach has a specific disadvantage. Its drawback is the chattering phenomenon, i.e. high frequency vibrations of the controlled system, which degrades the performance and may lead to instability. High order sliding modes (HOSMs) were created to remove the above restrictions hiding the switching in the higher derivatives of the sliding variable. These techniques consider a fractional power of the absolute value of the tracking error coupled with the sign function, this structure provides several advantages as simplification of the control law, higher accuracy and chattering prevention [17-19].

Sliding mode control design consists of two phases. In the first phase the sliding surface is to be reached (reaching mode), while in the second the system is controlled to move along the sliding surface (sliding mode). In fact, these two phases can be designed independently from each other.

Defining,

$$\sigma(e) = e = y - y_{ref} \tag{13}$$

as the sliding surface, we have that the continuous part of the sliding mode controller is given by,

$$u_{eq} = -\bar{g}(y,z)^{-1}(f_1(y,z) + \psi(y,z) + \phi(t) - \frac{dy_{ref}}{dt}) \tag{14}$$

Once on the surface, the dynamic response of the system is governed by $de/dt = 0$. To force the system trajectory to converge to the sliding surface in the presence of both model uncertainties and disturbances, with chattering minimization and finite-time convergence, the sliding trajectory is proposed as [19],

$$u_{dis} = -\bar{g}(y,z)^{-1}[\delta_1 e + \delta_2 \int_0^t sign(e)|e|^{1/p} d\tau] \tag{15}$$

where δ_1 and δ_2 are control design parameters. The final IHOSMC is given by,

$$u = u_{eq} + u_{dis} \tag{16}$$

Summarizing, the IHOSMC is composed by a proportional action, which has stabilizing effects on the control performance, and a high order sliding surface, which compensates the uncertain nonlinear terms to provide robustness to the closed-loop system. This behavior is exhibited because, once on the sliding surface, system trajectories remain on that surface, so the sliding condition is taken and make the surface and invariant set. This implies that some disturbances or dynamic uncertainties can be compensated while still keeping the surface an invariant set.

5. Numerical simulations

In this section numerical results for both case studies are presented. Dynamic optimization results are presented for the optimized trajectory corresponding to the desired reference for the control problem. The control performance with both robust control approaches is presented and compared.

5.1. Dynamic optimization results for case study.

The dynamic optimization for the case study, as described in Section 3.2, has been solved previously using NLP techniques [5,9]. We have obtained identical results and the resulting optimal trajectory for the crystallizer temperature can be approximated with the following profile,

$$\begin{aligned} T &= -2.6t + 322 \qquad 0 < t < 7 \\ T &= 303 \qquad\qquad\quad t > 7 \end{aligned} \tag{17}$$

The optimized trajectory allows to meet the constraints on the crystallizer, jacket temperature and concentration during the evolution of the closed-loop profiles. Based on the resulting optimized trajectory, the control design must track rapidly the temperature set point, such that a finite-time convergence is needed.

5.2. Modeling error compensation control.

Figure 1 shows the closed-loop performance under the control design based on MEC approach with finite-time convergence. Three sets of controller tuning parameters are shown.

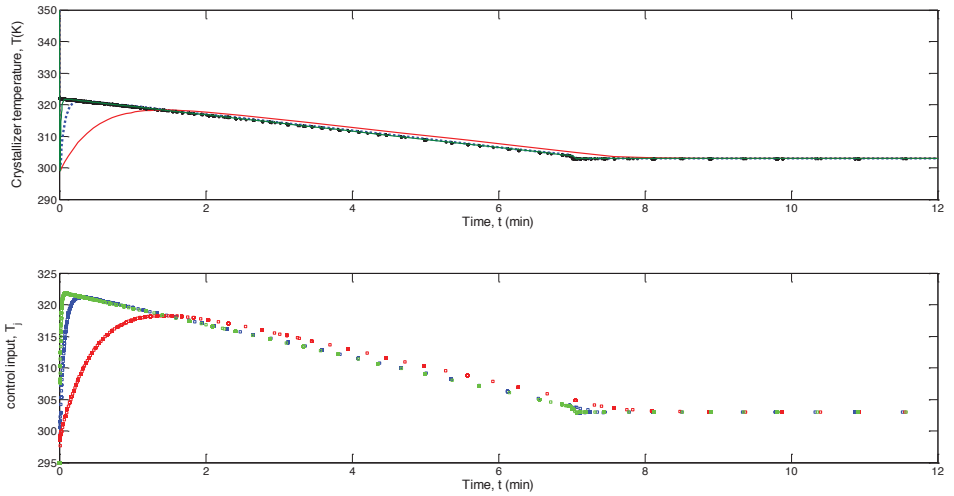

Fig. 1. (a) Controlled crystallizer temperature, y. (b) Jacket crystallizer temperature, u. Green line for $\tau_c = 0.1$, $\tau_e = 0.05$. Blue line for $\tau_c = 1$, $\tau_e = 0.2$. Red line for $\tau_c = 2.5$, $\tau_e = 0.5$.

It is noted that the MEC approach is able to track the desired optimal trajectory. The worst closed-loop performance is obtained for $\tau_c = 2.5$ and $\tau_e = 0.5$. For $\tau_c = 0.1$ and $\tau_e = 0.05$ and $\tau_c = 1$ and $\tau_e = 0.2$ it can be observed a very fast convergence to the desired optimized trajectory. The control input behaves very close to the controlled variable since the crystallizer wall is diathermy with the jacket crystallizer wall.

5.3. High-order sliding mode control.

Figure 2 shows the closed-loop performance under the control design based on high order SMC approach with finite-time convergence. Three sets of controller tuning parameters are also consider.

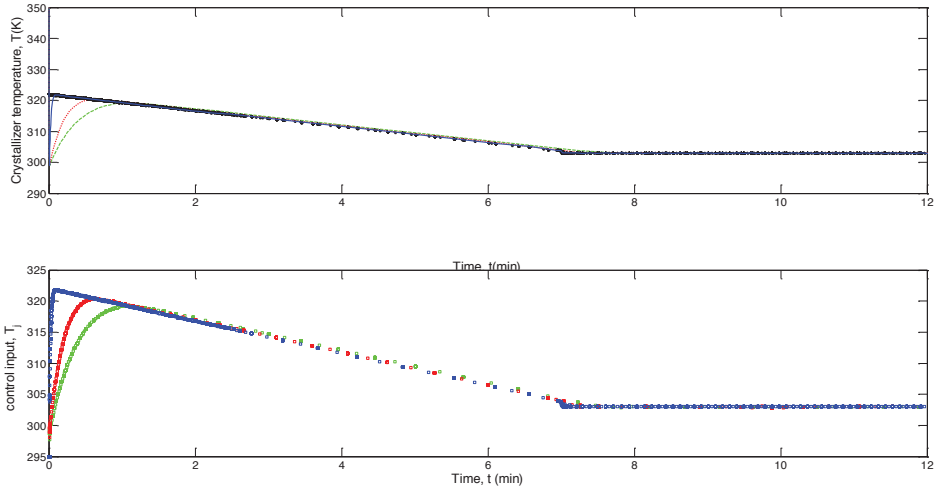

Fig. 2. (a) Controlled crystallizer temperature, y. (b) Jacket crystallizer temperature, u. Green line for $\tau_c = 0.1$, $\tau_e = 0.05$. Green line for $\tau_c = 1$, $\tau_e = 0.2$. Green line for $\tau_c = 2.5$, $\tau_e = 0.5$.

It can be observed from Figure 2 that the SMC approach, as the MEC approach, is able to track the desired optimal trajectory. In this case, the worst closed-loop performance is obtained for $\delta_1 = \delta_2 = 0.05$. For $\delta_1 = \delta_2 = 0.1$ and $\delta_1 = \delta_2 = 1$ it can be observed a very satisfactory closed-loop performance with a quickly convergence to the desired optimized trajectory.

For both control designs, it is noted that both controllers can successfully track the desired optimal trajectory with a very similar closed-loop performance and control effort. This can be related to the finite-time convergence properties of both control approaches.

6. Conclusions

In this study, the dynamic optimization and the robust feedback control of a general class of batch crystallizers models has been presented. The dynamic optimization problem leads to a NLP optimization problem, which is solved with the software *dynopt,* which uses an algebraic parameterized approach that is solved using a sequential quadratic programming algorithm. The proposed robust control approaches are derived considering two features of batch crystallization processes: (i) a desired finite time convergence, and (ii) model uncertainties. The robust control approaches are the modelling error compensation with finite-time convergence and the high-order SMC approach. A case study of a non-isothermal batch crystallizer was used. Simulation results show that both the MEC and high-order SMC controllers leads to a good closed-loop performance.

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